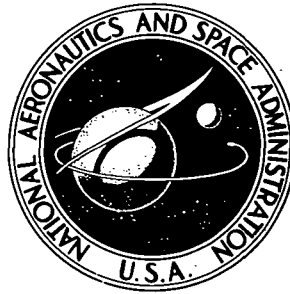


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## SCANNING ELECTRON MICROSCOPE STUDY OF POLYTETRAFLUOROETHYLENE SLIDING ON ALUMINUM SINGLE CRYSTALS

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16. Abstract  <p>Friction experiments were conducted in air with polytetrafluoroethylene (PTFE) sliding on aluminum single crystals. Mechanical scoring of the crystals with (110) and (100) orientations was observed with a single pass of the PTFE slider. No scoring was observed on the (111). The degree of scoring of the crystals is related to the hardness, with the hardest surface (111) showing no damage and the softest surface (110) showing the most severe scoring. Scoring is caused by work-hardened pieces of aluminum which, as a consequence of the adhesion between PTFE and aluminum, were pulled out of the bulk and became embedded in the PTFE polymer.</p>					
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# SCANNING ELECTRON MICROSCOPE STUDY OF POLYTETRAFLUOROETHYLENE

## SLIDING ON ALUMINUM SINGLE CRYSTALS

by William A. Brainard and Donald H. Buckley

Lewis Research Center

### SUMMARY

Friction experiments were conducted in air with polytetrafluoroethylene (PTFE) riders sliding on aluminum single crystals. The single crystals were in the (111), (110), and (100) orientations. Wear tracks were generated on the single crystals at loads of 100 and 200 grams. Following running, the wear tracks were observed in the scanning electron microscope for evidence of damage to the metal surfaces.

It was observed that scoring of the (110) and (100) single crystal planes occurred with single passes of the PTFE rider over the surface. No scoring was observed on the (111) plane even with repeated passes. The scoring was the result of pieces of aluminum being pulled out of the bulk metal and embedding in the soft polymer rider. These embedded pieces served to act as abrasives and generated plow tracks in the soft metal surfaces.

The degree of scoring was related to the hardness of the individual single crystals. For the hardest single crystal (111), no scoring was observed. For the softest crystal (110), the scoring was most severe. For the (100), which was intermediate in hardness, the scoring was intermediate in degree.

### INTRODUCTION

In the field of lubrication, the polymeric materials have found application in many areas where, because of low friction properties, they function as solid lubricant materials in a mechanical component such as a ball-bearing cage. Polytetrafluoroethylene (PTFE), one of these polymers, has been widely studied and many attempts have been made to utilize this material because of its attractive physical and chemical properties (refs. 1 to 3). Earlier studies in this laboratory have shown that adhesive transfer films of PTFE develop during sliding as well as in simple static contact for a wide range

of materials. Specifically, adhesive transfer of PTFE was confirmed to have occurred by Auger Emission Spectroscopy for PTFE contacting clean metals (e.g., tungsten and aluminum), passive metals such as gold, as well as glass and oxide surfaces (ref. 4). Although transfer was observed for all these materials with apparent disregard for chemical constitution, evidence does suggest that the bonding of the polymer to the metal can be extremely strong. Studies in the field ion microscope where PTFE was used to contact a clean tungsten field ion tip, confirmed, on the atomic level, the transfer of the polymer to the metal surface. It also provided a measure of the strength of the polymer to metal bonding (ref. 5) which approached that of clean metals in contact. As a consequence of the strong adhesion of PTFE, it was observed that sliding on an aluminum surface produced severe metallic disruption. Specifically, the PTFE rider "machined" the aluminum surface cutting deep grooves in the metal.

It is the objective of this investigation to study the sliding of PTFE on aluminum surfaces under atmospheric conditions to determine the nature of the "machining" effect and also to characterize this effect by microscopic examination. To achieve this and, PTFE riders were drawn across single crystal aluminum surfaces and the resulting wear tracks examined by scanning electron microscopy.

## MATERIALS

The aluminum face-centered-cubic single crystals were commercially purchased. The orientations were (111), (110), and (100) and the diameter was 2.5 centimeters. The purity of the aluminum was 99.999 percent. The surfaces were prepared by hand polishing on wet silicon carbide paper down to 600 grit. They were then diamond paste polished and finally polished with 0.3 micron aluminum oxide. Following mechanical polishing, the crystals were electropolished in phosphoric acid to remove the worked surface layer.

The PTFE riders were machined from high purity research grade PTFE bar stock. A radius of 1.5 millimeters was cut on each rider. The radiused region was prepared by filing with a fine pattern makers file. Following radiusing, the riders were rinsed with absolute ethyl alcohol and dried.

## APPARATUS AND PROCEDURE

The apparatus used to draw the single crystal under the rider is shown in figure 1. It is a modified microscratch hardness tester to which a drive motor with a gear reduction head was attached to provide uniform motion of the single crystals. In this study a

constant speed of 1.4 millimeters per minute was used. The crystals were oriented on the apparatus such that sliding took place in the  $\langle 100 \rangle$  direction on the (100) and (110) planes and in the  $\langle 110 \rangle$  direction on the (111) planes. One centimeter wear tracks were generated with each pass.

The PTFE rider was mounted in an arm above the single crystal. Loading was accomplished by the application of dead weights directly over the rider. Loads of 100 and 200 grams were used. A freshly prepared rider was used for each experiment. The rider is mounted above the crystal in an arm which is restrained by a strain gage assembly. Frictional forces are measured by the strain gage during sliding. All the experiments were conducted in air at approximately 20° C with a relative humidity of near 20 percent.

Following the friction experiments, the single crystals were inserted into a scanning electron microscope (SEM) for examination of the generated wear scars. The SEM used for the examination was a field emission electron source type with a television scanning rate of 15 pictures per second. This unit has the advantage that the electron beam at the very fast scanning rates at all magnifications is less likely to cause damage to the polymer than a slower scanning SEM. Thus, the transfer film could be observed directly without the necessity of either metallizing or replicating.

All micrographs were taken at a tilt angle of 45° with the tilt axis being horizontal on the micrographs. The magnifications given are all nominal.

## RESULTS

Sliding wear tracks were generated on each of the three single crystal surfaces under loads of 100 and 200 grams. Single passes as well as multipasses over the same track were made. In all cases, large disordered areas of PTFE were observed adhering to the aluminum surfaces at the initial contact area. Extending out of these areas were linear fibers of PTFE parallel to the surface. These were oriented in the direction of sliding. The amount of material transferred at the initial contact as well as extending from it were load-dependent. This is shown in figure 2. The starting friction coefficients in these areas were quite high (0.20 to 0.25). The friction dropped, however, rapidly once sliding progressed. The initial disordered areas could be a result of the "loose" fragments of PTFE on the riders as a result of the abrasive preparation by filing. These fragments are readily observable in the SEM micrographs shown in figure 3. The strands are probably subject to some static charging in these micrographs and in reality are not all normal to the surface as shown.

The width of the track and the amount of PTFE remaining after a single pass for loads of 100 and 200 grams on a (111) aluminum surface are shown in figure 4(a). Fig-

ure 4(b) shows the tracks generated by a single pass at 100-gram loads for the (110) and (100) planes.

The contrast given in the SEM by the PTFE fibers on the surface is dark when the fibers are contacting the metal surface; for strands which are above the surface, however, the contrast is white. At regions on the surface where the wear track traversed a depression, the strands were observed to be pulled across the depression in an almost equally spaced parallel manner. These observations confirm the results obtained by replication techniques used by Steijn, (ref. 6). These fibers changed their contrast from dark on one side of the depression, to white where they were suspended off the surface over the depression and again dark on the other side. This is illustrated in the stereo-pair shown in figure 5.

On all the single crystals during the first 2 to 3 millimeters of the wear track large particles of PTFE were observed adhered to the metal surface with polymer strands extending outward parallel to the direction of sliding (fig. 6). Fine fiber-like ribbon, as well as large particles of PTFE, were observed on the (110) plane near the starting points (fig. 7). The ribbons were so thin that at some points the wear track could be seen through them.

The friction coefficients for the three crystals were in the range of approximately 0.06 to 0.20 during sliding. The friction on the (111) surface was generally the lowest ( $0.06 \leq f_k \leq 0.12$ ) with the range for the (100) and (110) plane being somewhat higher ( $0.12 \leq f_k \leq 0.20$ ).

On the (111) plane, no damage to the aluminum surface was observed with a single pass of the rider at either 100- or 200-gram loads. Further, repeated passes over the same track did not damage the metal surface. On the (100) and (110) planes fine score marks were observed on the aluminum surface after single passes at 200 grams. At the lightest load tested (100 grams), no scoring was observed on the (100) plane but scoring was observed on the (110) plane. Figure 8 shows micrographs showing scoring of the metal taken near the end of the wear tracks on the (110) plane for 100- and 200-gram loads. The higher magnification micrograph taken for the 200-gram load shows areas where pieces of aluminum have been plucked out of the bulk (see fig. 8(b)).

For PTFE sliding on the (100) plane, although no scoring was observed for a single pass at 100-gram load, fine score marks were observed after three passes over the same wear track as shown in figure 9. On the (110) plane, repeated passes resulted in extensive scoring for a 200-gram load. In general, score marks on both planes were found to terminate with a fragment of PTFE which had been pulled out of the bulk as shown in figure 10.

## DISCUSSION

The results with PTFE sliding on aluminum have shown that surface damage to the metal can occur as a result of sliding contact. The damage will occur even in air where the aluminum has a natural protective coating of hard oxide ( $\text{Al}_2\text{O}_3$ ). The generation of the transfer film of the polymer onto the metal surface has been shown to be a consequence of drawing of long strands of PTFE out of the bulk and orientation of these strands parallel to the direction of sliding. The drawing of strands and their orientation have been observed by others using both optical microscopy and surface replication techniques with the electron microscope (refs. 6 and 7). The drawings of these strands from the bulk during sliding requires that the PTFE strand be bonded to the metal surface with sufficient strength to anchor it. Makinson and Tabor, during their studies of PTFE sliding on metals, suggested that the bonding of the PTFE to the metal surface must be quite strong, and this strong bonding was later confirmed by adhesion experiments in the field ion microscope (ref. 7). An obvious question is raised; is the bonding of the PTFE to the metal surface sufficiently strong to initiate transfer of the metal to the polymer and then be responsible for the scoring observed?

It is tempting to ascribe the scoring of the aluminum to the hard oxide on the aluminum surface becoming embedded in the soft bulk of the polymer, thus acting like an abrasive cutting tool over the surface. This does not seem to be the case for two reasons. First, experiments conducted in vacuum with a clean aluminum surface which had all the oxide removed by argon ion bombardment still showed the pronounced machining of the surface. Second, no scoring was observed on the (111) plane and the  $\text{Al}_2\text{O}_3$  if responsible would also be equally likely to score this surface.

Yet it does appear that hard materials embedded in the polymer are responsible for the scoring observed. For example, when the PTFE rider was abraded with silicon carbide paper prior to sliding, thus embedding some loosened carbide particles on the PTFE surface, scoring was observed on all planes in a manner very similar to the results described earlier. Further, if small fragments of diamond were not removed from the aluminum surface after diamond paste polishing, scoring was also indiscriminately observed at those points in the wear tracks where the PTFE slid over a diamond particle. The hard carbide or diamond particles would be pushed along by the rider plowing the softer metal. At the point when the prow of the track had accumulated a sufficient buildup of metal in front of the rider to prevent any further plowing, fracture occurred in the polymer causing a large fragment of PTFE to break off thus halting the plowing at that point. This process is shown nicely in figure 11, where a diamond particle was pushed along the surface until the buildup of material in front of it was sufficiently large to stop the process leaving behind a large fragment of PTFE. This break off of a fragment of PTFE is identical to those shown in figure 10.

Thus it appears as if scoring of the aluminum is caused by hard particles lodged in the PTFE. Discounting  $\text{Al}_2\text{O}_3$  as the source of the hard particles, it seems likely that fragments of aluminum are being pulled out of the bulk during sliding and embedded in the PTFE rider. With continued sliding, these aluminum fragments become work hardened and cause scoring. This is confirmed by the observation in the SEM, of aluminum fragments embedded in the PTFE rider after running (fig. 12).

The differences observed in the scoring behavior of the three aluminum single crystals appears to be correlated with the different mechanical properties of the three single crystals. Microhardness measurements on the three surfaces showed the order of hardness to be :  $(111) > (100) > (110)$ . The protection afforded by the oxide breaks down at very light loads because of the hard brittle nature of the oxide (ref. 8). Thus, the PTFE penetrates to the nascent metal and, as a consequence of adhesion and the localized stress, plucks some of the metal from the surface and these metal fragments are pushed along with the slide. As sliding continues, these pieces of metal become strain hardened until they are harder than the softer metal surface and can then score the underlying aluminum. On a (111) surface where the hardness is the highest, this process does not occur. On the (110) plane which is the softest, scoring is most severe. On the (100) plane which is intermediate, between the (111) and (110), scoring is intermediate in degree.

## SUMMARY OF RESULTS

Sliding friction experiments with polytetrafluoroethylene (PTFE) sliding on (111), (110), and (100) aluminum single crystals in air showed the following results:

1. Mechanical scoring of the aluminum single crystals occurs in air for the (110) and (100) planes. No scoring was observed on the (111) plane.
2. The scoring of the single crystal surfaces is related to the hardness. The hardest crystal (111) showed no scoring; the softest crystal (110) showed the severest scoring; and the crystal intermediate in hardness (100) showed scoring intermediate in degree.
3. The scoring is the result of pieces of aluminum being pulled off the single crystal surface. These metal pieces lodge in the soft polymer and, with work hardening, serve to plow the softer aluminum substrate.

Lewis Research Center,

National Aeronautics and Space Administration,

Cleveland, Ohio, April 11, 1973,

502-01.

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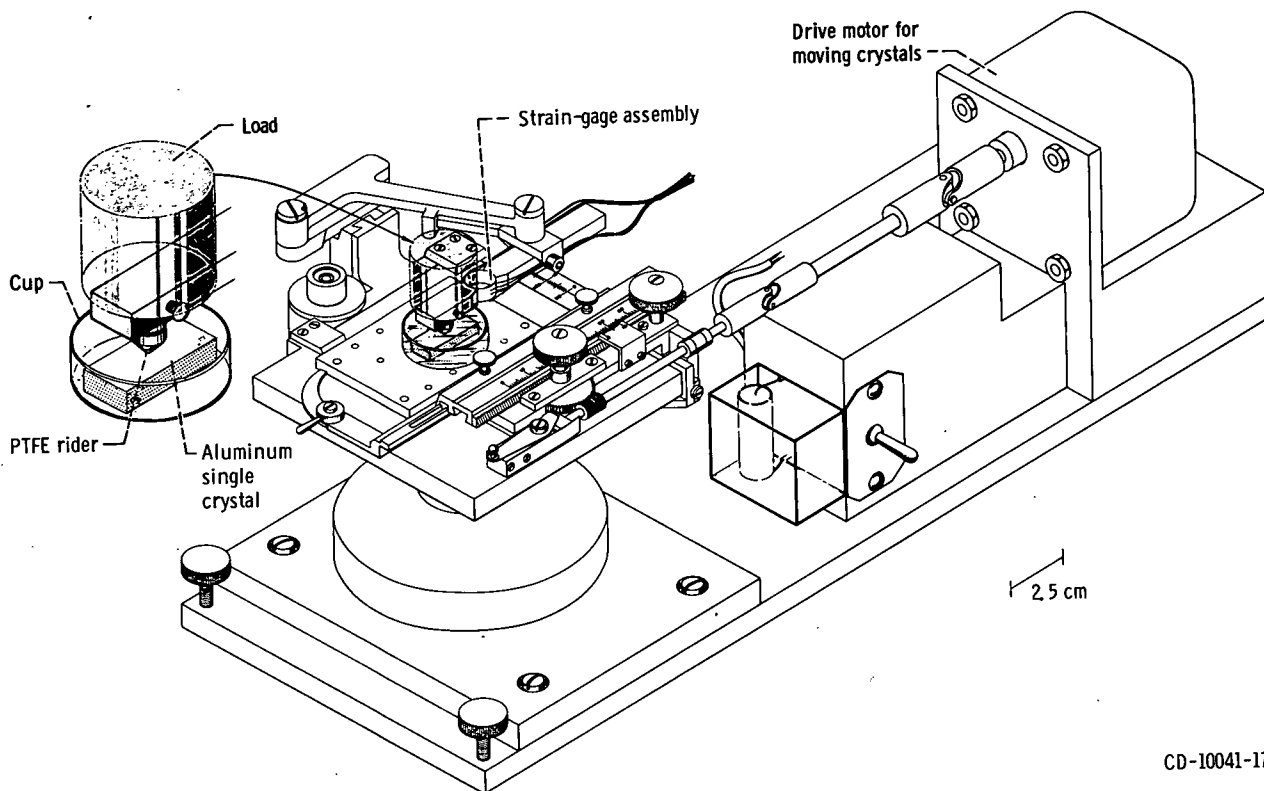
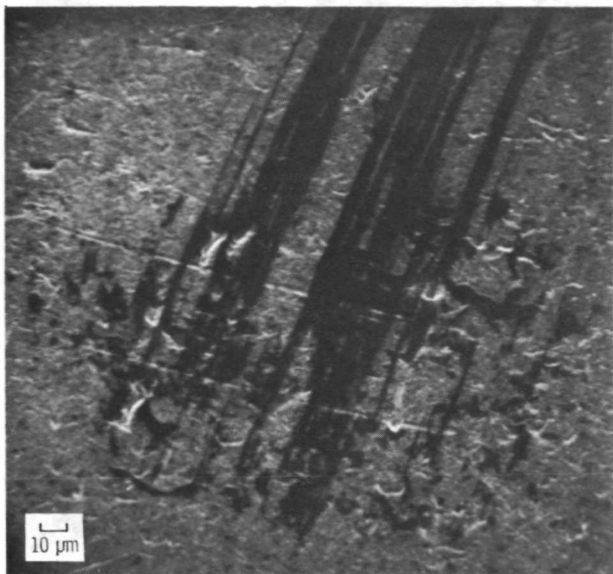
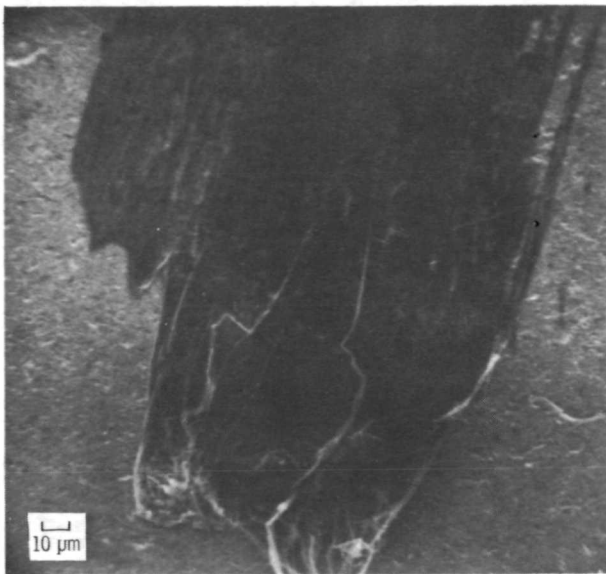


Figure 1. - Sliding friction apparatus.



(a) (111) plane; 100 grams.



(b) (111) plane; 200 grams.

Figure 2. - Start of PTFE-aluminum wear track.

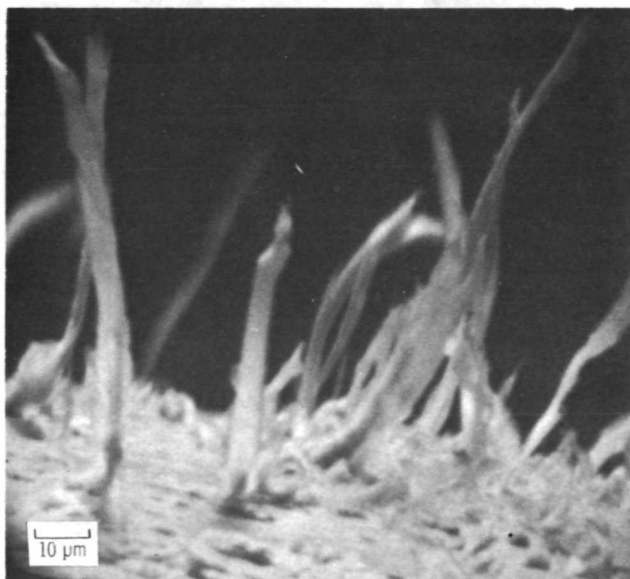
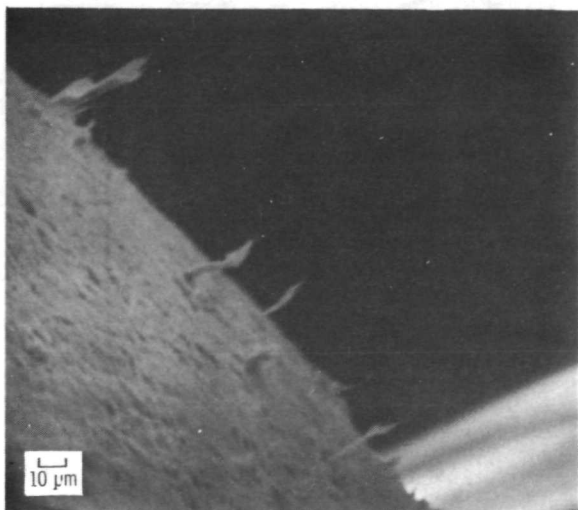
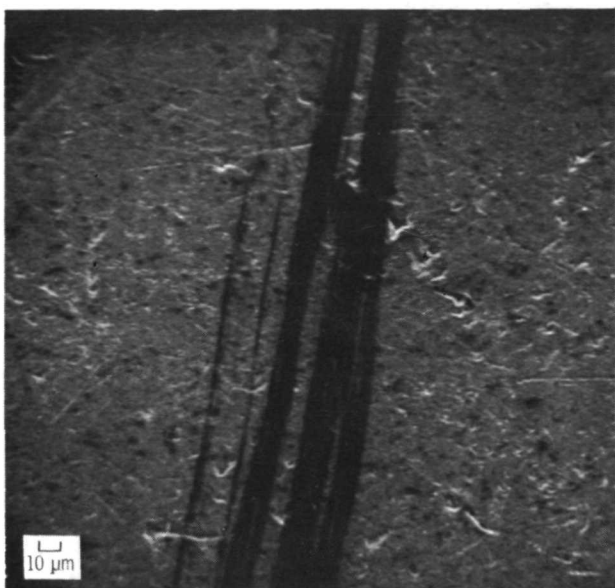
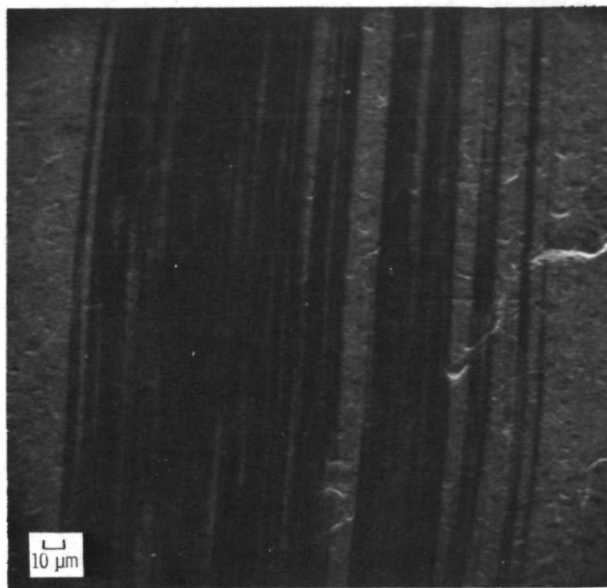


Figure 3. - Surface of PTFE slider prior to sliding.

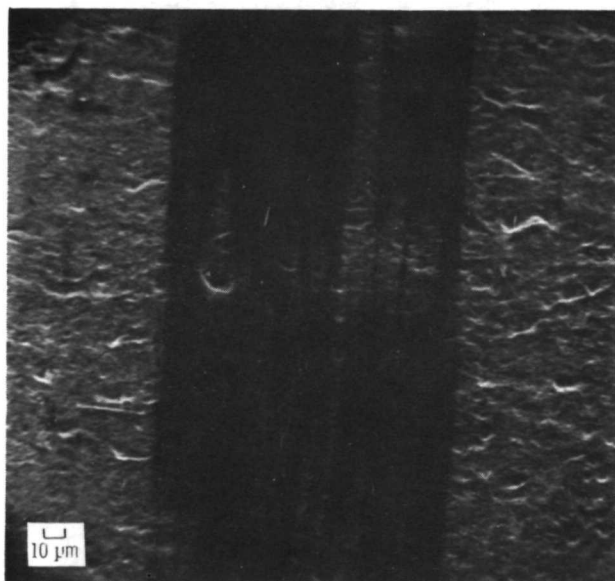


(a-1) 100-gram load.

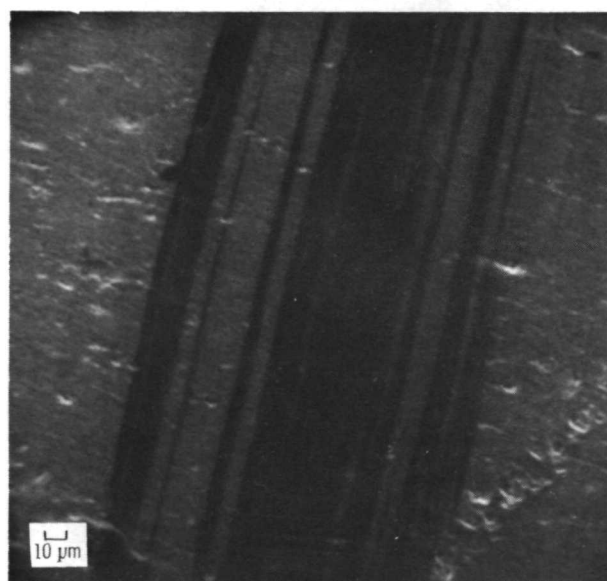


(a-2) 200-gram load.

(a) (111) plane.



(b-1) (110) plane.



(b-2) (100) plane.

(b) 100-gram load.

Figure 4. - PTFE wear tracks on aluminum surface.

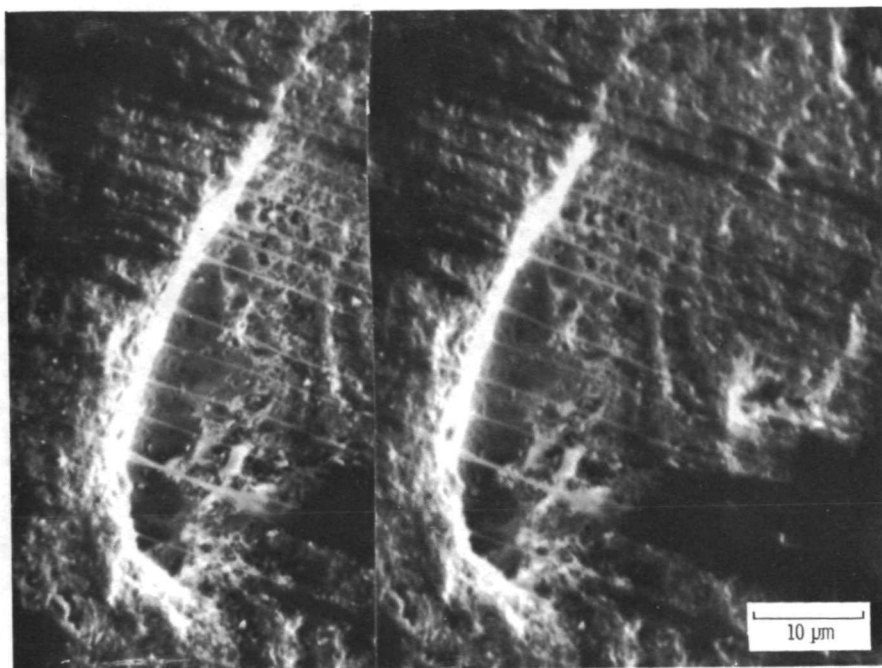


Figure 5. - Wear track on (111) surface; 200-gram load; stereo pair.

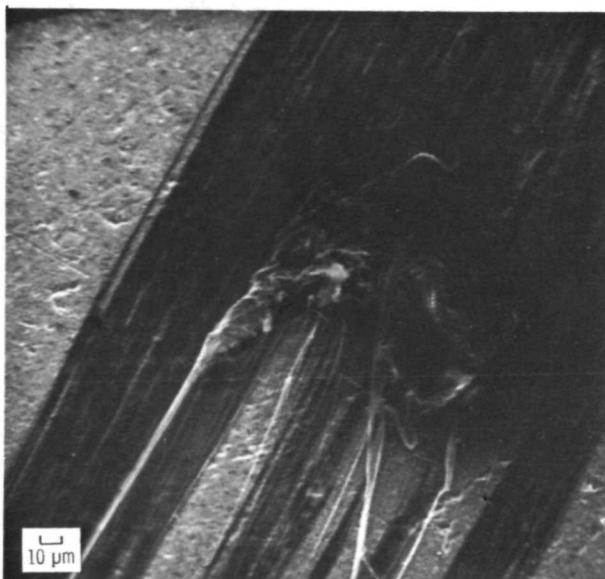
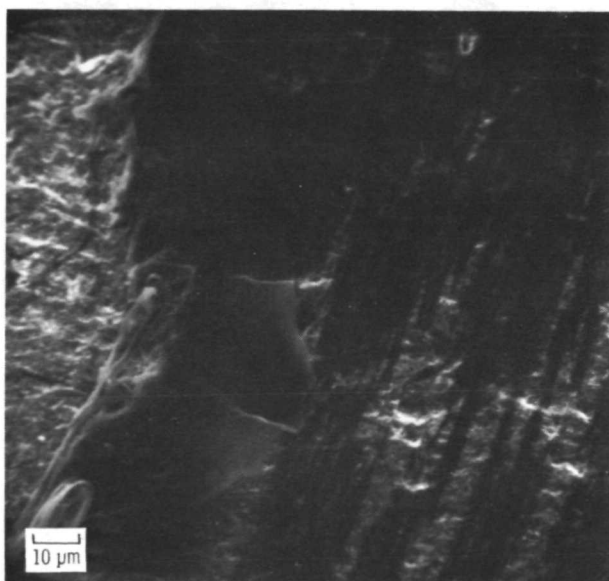


Figure 6. - Wear fragments of PTFE in wear track.

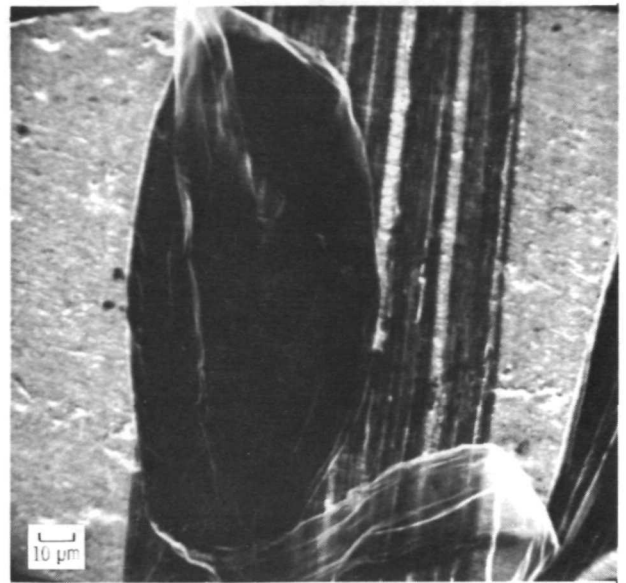
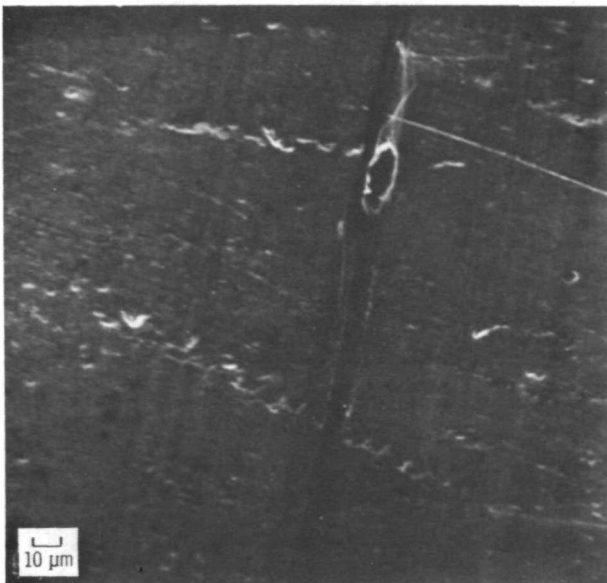
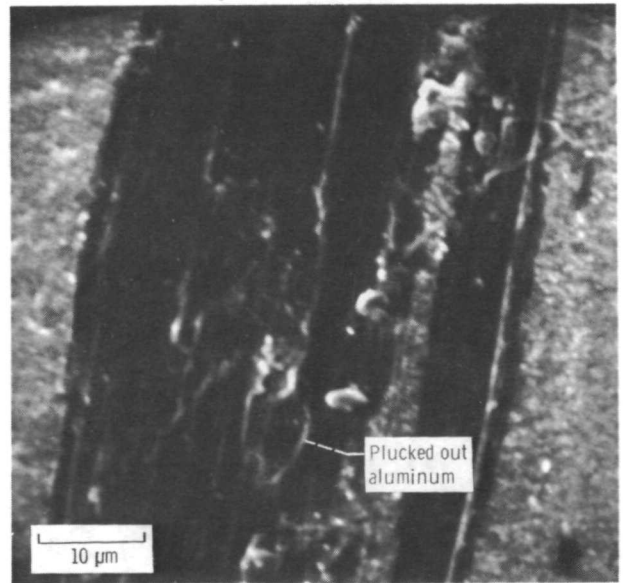


Figure 7. - Wear tracks showing ribbon-like PTFE debris.



(a) 100-gram load.



(b) 200-gram load.

Figure 8. - Wear track on (110) plane after single PTFE slider pass.

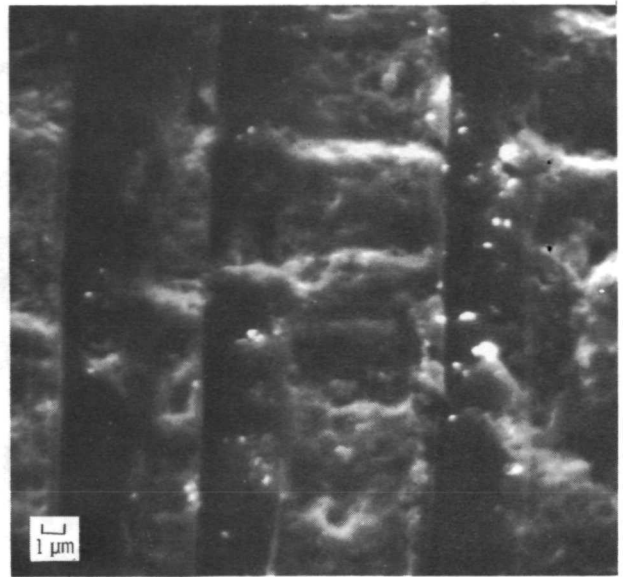
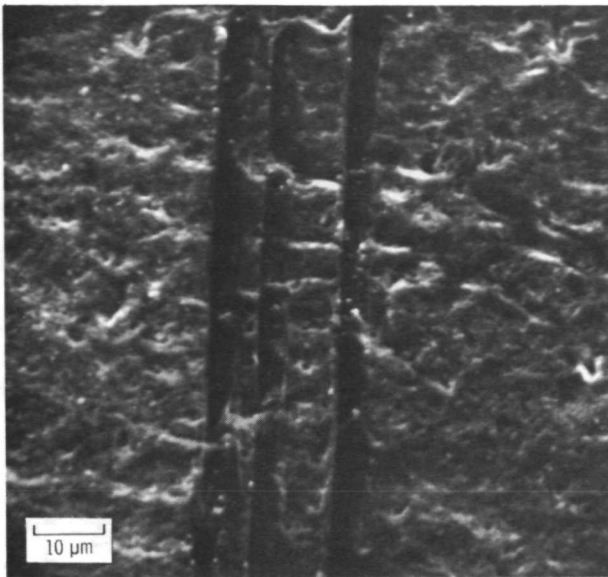
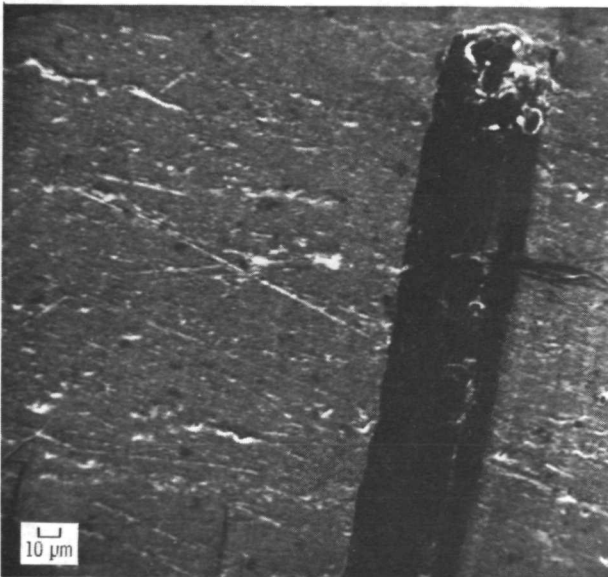
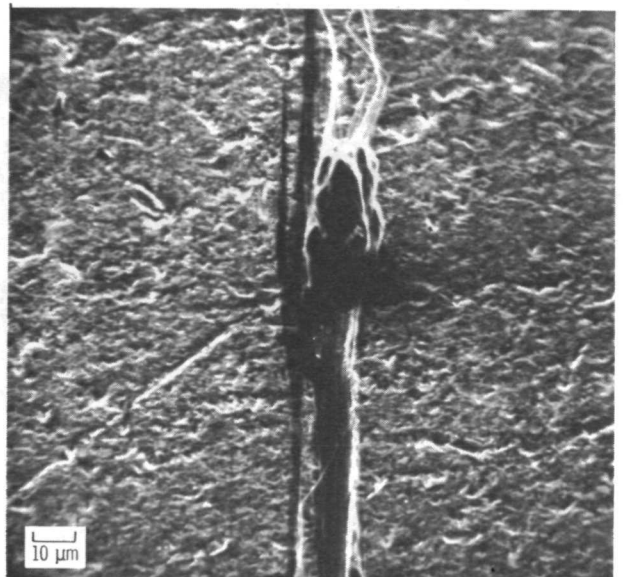


Figure 9. - PTFE wear tracks on (100) surface; 100-gram load.



(a) (110) plane.



(b) (100) plane.

Figure 10. - PTFE fragments at end of score marks.

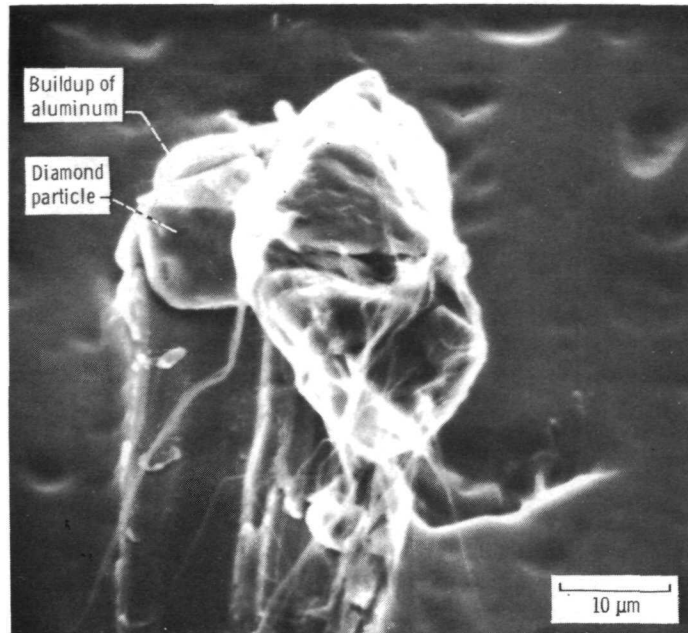


Figure 11. - Termination of score caused by hard diamond particle.

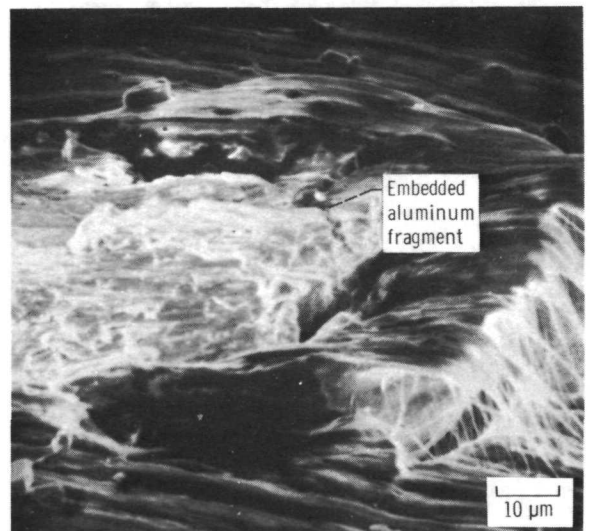
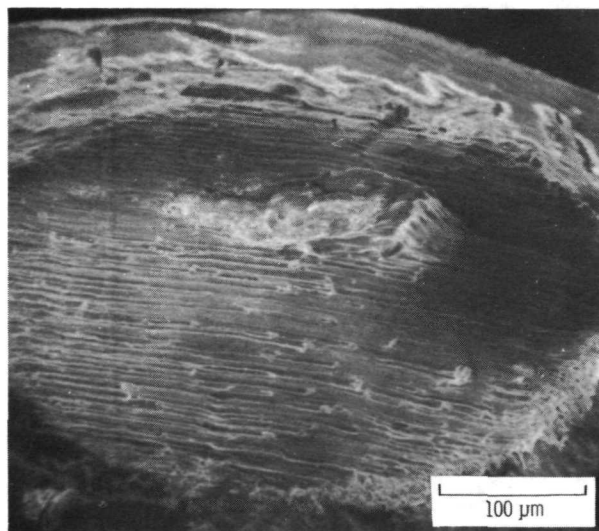


Figure 12. - PTFE-rider wear scar showing lodged metal fragment. Run on (110) surface; single pass; 200-gram load.



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